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IS 13193 (1992): Polyalkylene terephthalates (PET and PBT) for moulding and extrusion [PCD 12: Plastics]

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Indian Standard

POLYALKYLENE TEREPHTHALATE (PET AND PBT) FOR MOULDING AND EXTRUSION — SPECIFICATION

UDC 678·644·142

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1992 Price Group 7

Plastics Sectional Committee, PCD 12

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Polyalkylene terephthalate are polycondensation products or esterification between a dibasic acid or its derivatives with a diol. Polyethylene terephthalate (PET) and Polybutylene terephthalate (PBT) which are the most widely used polyalkylene terephthalate materials are the products derived by the esterification of dimethyl terephthalate (DMT) or purified terephthalic acid (PTA) with monoethylene glycol and 1,4-butane-diol respectively. PET and PBT are also known as 'saturated or thermoplastic polyesters'.

PET and PBT are grouped under the family of 'engineering thermoplastics' due to their good combination of properties suitable for high performance usage. Their ability to be modified by simple techniques by addition of either reinforcing agents or speciality fillers/additives makes them popular for a diverse range of applications in packaging, automotive, electrical, electronic, telecommunications, appliances, business machines, railways and many other industries.

Thermoplastic polyester (mainly PET) are converted into oriented products by stretching the molecular chains in mono or biaxial direction during processing. The orientation enhances the mechanical, barrier and optical properties or the end products. Thermal resistance also improves in some cases. Monofilaments and strappings represent the monoaxially oriented products whereas the films, bottles/cans, etc, represent the biaxially oriented products.

This standard contains clauses **5.2.1**, **5.3.1**, **5.4.2**, **5.5.2**, **5.6.1**, **5.7** and **6.1** which call for an agreement between the purchaser and the supplier.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 DECEMBER 1993
TO
IS 13193 : 1992 POLYALKYLENE TEREPHTHALATE (PET AND PBT) FOR MOULDING AND EXTRUSION — SPECIFICATION

[*Page* 2, *Table, Sl No.* (iii), *col* 2] — Substitute '25 ± 0.1°C' *for* '25 ± 0.01°C'.

(*Page* 2, *clause* **5.3.1**) — Read as follows in place of the existing:

'**5.3.1** The requirements for diethylene glycol (DEG) content, sulphated ash percent by mass (Ash content) and carboxyl group content shall be as agreed to between the purchaser and the supplier.'

(*Page* 2, *clause* **5.4.3**) — Read as follows in place of the existing:

'**5.4.3** The moisture content during processing shall not be more than 0.004 percent when tested as prescribed in Annex B'

(*Page* 4, *clause* **A-3.8**) — Substitute '25 ± 0.1°C' in place of '25 ± 0.05°C'.

(PCD 12)

Reprography Unit, BIS, New Delhi, India

Indian Standard

POLYALKYLENE TEREPHTHALATE (PET AND PBT) FOR MOULDING AND EXTRUSION — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and tests for polyalkylene terephthalate materials for moulding and extrusion.

1.2 This standard is intended to be used for identification and characterization of polyalkylene terephthalate materials on the basis of basic polymer parameters and recommended end-uses.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
2828 : 1964	Glossary of terms used in plastic industry
4905 : 1968	Methods for random sampling
8543 (Part 1/ Sec 2) : 1979	Methods of testing plastics: Part 1 Characterization of polymer structure and size, Section 2 Determination of density of solid plastics
8543 (Part 3/ Sec 2) : 1978	Methods of testing plastics: Part 3 Preparation of test specimens, Section 2 Injection moulded test specimens
8543 (Part 4/ Sec 1) : 1984	Methods of testing plastics: Part 4 Short term mechanical properties, Section 1 Determination or tensile properties
13411 : 1992	Glass reinforced polyester dough moulding compound
13360 (Part 6/ Sec 1) : 1992	Plastics — Methods of testing: Part 6 Thermal properties, Section 1 Determination of vicat softening temperature of thermoplastics materials
13360 (Part 6/ Sec 6) : 1992	Plastics — Methods of testing: Part 6 Thermal properties, Section 6 Determination of flammablity by oxygen index
13360 (Part 6/ Sec 10) : 1992	Plastics — Methods of testing: Part 6 Thermal properties, Section 10 Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers
12252 : 1987	Polyalkylene terephthalates (PET & PBT) for their safe use in contact with foodstuffs, pharmaceuticals and drinking water

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 2828 : 1964 shall apply.

4 TYPES

There shall be two types of polyalkylene terephthalate materials, namely, polyethylene terephthalate (PET) and polybutylene terephthalate (PBT).

5 REQUIREMENTS

5.1 Description

The material shall be thermoplastic (saturated) polyester, produced by the catalytic condensation of DMT/PTA at high temperature and reduced pressure with monoethylene glycol and 1,4-butane-diol for making PET and PBT types respectively.

5.2 The material shall be in granular form.

5.2.1 Any modification through reinforcements and by addition of fillers, colours, flame retardants, heat stabilizers, lubricating agents, etc, and its requirements based on end use functionality which may be necessary in some specific applications shall be agreed upon between the purchaser and the supplier.

5.3 The material shall comply with the requirements laid down in Table 1 when tested according to the methods mentioned corresponding to each requirements under col 5 and 6 of the table.

Table 1 Requirements for Polyalkylene Terephthalates (PET & PBT) (Unfilled Natural Colour)

(*Clause* 5.3)

Sl No.	Characteristic	Requirement for Type		Method of Test, Ref to	
		PET	PBT	Indian Standard	Annex of this Standard
(1)	(2)	(3)	(4)	(5)	(6)
i)	Density, g/cm^3	1·38 ± 0·02	1·30 ± 0·02	8543 (Part 1/Sec 2) : 1979	—
ii)	Melting point, °C	255 to 265	223 to 225	13360 (Part 6/Sec 10) : 1992	—
iii)	Relative viscosity at 25±0·01°C	1·6 to 2·2	1·6 to 2·4	—	A
iv)	Heat distortion, temperature, °C, *Min*			Annex B of 13411 : 1992	—
	a) at 18·5 kg/cm^2	70	60		
	b) at 4·6 kg/cm^2	72	165		
v)	Vicat softening point at 1 kg, °C, *Min*	78	210	13360 (Part 6/Sec 1) : 1992	—
vi)	Tensile strength at break, MPa, *Min*	55	52	8543 (Part 4/Sec 1) : 1984	—
vii)	Elongation at break, percent	50 to 200	50 to 200	8543 (Part 4/Sec 1) : 1984	—
viii)	Impact strength, Izod (notched), kj/m^2, *Min*	2·0	2·0	Annex E of 13411 ; 1922	—
ix)	Cross breaking strength, at maximum load, MPa, *Min*	80	75	Annex F of 13411 : 1992	—
x)	Moisture content, percent by mass, *Max*	0·01	0·01	—	B

5.3.1 The requirements for diethylene glycol (DEG) content, sulphated ash percent by mass and carboxyl group content shall be as agreed to between the purchaser and the supplier.

5.4 Special Requirement for Material for Stretch Blow-Moulding

5.4.1 The material shall be of PET type.

5.4.2 *Relative Viscosity*

The relative viscosity when tested in accordance with the method prescribed in Annex A shall be as per agreement between the purchaser and the supplier. The most common range of relative viscosity is from 1·75 to 1·85.

5.4.3 *Moisture*

The moisture content shall not be more than 0·004 percent when tested as prescribed in Annex B.

NOTE — Continuous drying system operating with dehumidified hot air with dew point of minimum −30°C is essential to reduce the moisture level to about 0·004 percent for processing PET.

5.5 Special Requirement for Material for Making Films, Monofilaments Strappings, Sheet, etc

5.5.1 The material shall be of PET type. PBT may also be used.

5.5.2 *Relative Viscosity*

The relative viscosity when tested in accordance with the method prescribed in Annex A shall be as per the agreement between the purchaser and the supplier. The most common range of relative viscosity is from 1·60 to 1·75.

5.5.3 *Moisture*

The material shall also conform to the requirement as mentioned in 5.4.3.

5.6 Special Requirement for Material for Flame Retardent Applications

5.6.1 The material shall conform to ratings FV-0, FV-1, FV-2, etc, and to oxygen index values as per the end-use requirements and agreements between the purchaser and the supplier when tested in accordance with the method prescribed in Annex C and IS 13360 (Part 6/Sec 6) : 1992 respectively.

5.7 There may be other requirements necessary for specific applications. These may be specified by agreement between the purchaser and the supplier.

5.8 Contact with Foodstuffs, Pharmaceuticals and Drinking Water

When used in contact with foodstuffs, pharmaceuticals and drinking water, its requirements with respect to material shall also be met as per IS 12252 : 1987.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed in bags/containers with suitable liner as agreed to between the purchaser and the supplier, in a manner so as to provide protection against ingress of moisture and to facilitate easy handling.

6.2 Marking

Each package shall be clearly marked with the following information:

a) Name and Type of the material;
b) Net mass of the material;
c) Indication of the source of manufacture and recognized trade-mark, if any;
d) Batch number in code or otherwise to enable the lot of manufacture to be traced from records; and
e) Month and year of the manufacture.

6.2.1 The packages may also be marked with the Standard Mark.

7 SAMPLING

The method of drawing representative sample of the material from a lot and the criteria for conformity of the material to the requirement of this standard shall be as prescribed in Annex D.

8 TEST SPECIMENS

8.1 Preparation of Test Specimen

Injection moulded test specimen shall be prepared as prescribed in IS 8543 (Part 3/ Sec 2) : 1978.

ANNEX A

[*Clauses* 5.4.2 *and* 5.5.2, and *Table* 1, *Item* (iii)]

DETERMINATION OF RELATIVE VISCOSITY

A-1 PRINCIPLE

Mesurement of the times of flow of the solvent and a solution of resin at a concentration of 0·005 g/ml at a 25°C by conventional methods; calculation of the relative viscosity from these measurements. Density difference and kinetic energy corrections are small in this method and are not applied.

A-2 SOLVENTS

> **Warning** — The solvents are hazardous. Avoid contact with the skin and inhalation of vapours.

A-2.1 Phenol/1,2-Dichlorobenzene

Weigh out 1 part by mass phenol (Analytical grade) and dissolve in 1 part by mass 1, 2-dichlorobenzene (analytical grade). Work to an accuracy of 1 percent or better in the weighings.

A-2.2 *m*-Cresol

The compound shall meet the following requirements:

Appearance	: Clear and colourless;
Content of *m*-cresol	: 99 percent (*m/m*), *Min*;
Content of *o*-cresol	: 0·3 percent (*m/m*), *Max;* and
Content of water	: 0·13 percent (*m/m*), *Max*.

NOTE — A solvent of the required purity can be obtained by distillation of chemically pure *m*-cresol, preferably in vacuo., pressure compensation being effected with nitrogen in order to avoid oxidation. The purity of the solvent may be checked by gas chromatography.

A-2.3 Storage of the Solvent

Protect the solvents from light by storing them, for example, in stoppered brown glass bottles.

Determine the flow time of the solvents at least once each day that they are used (*see* **A-4.6**). If the flow time of a solvent differs by more than 1 percent from the initial value, reject the solvent.

A-3 APPARATUS

A-3.1 Mill, to reduce the sample to grain size of about 0·5 mm.

A-3.2 Volumetric Flask, of capacity 100 ml, fitted with a ground glass stopper.

A-3.3 Erlenmeyer Flask, of capacity 150 ml, fitted with a ground glass stopper.

A-3.4 Burette, graduated in division of 0·1 ml.

A-3.5 Steam Bath, or other means to heat the contents of a flask to 90°-100°C.

A-3.6 Oil Bath, capable of being controlled at 135°-140°C or other means to heat the contents of a flask to that temperature range.

A-3.7 Magnetic Stirrer, or other equipment to continuously agitate the contents of a stoppered flask.

A-3.8 Thermostatic Bath, capable of being controlled at 25 ± 0·05°C.

A-3.9 Viscometers, suspended-level Ubbelohde type. The essential dimensions of the viscometers are shown in Fig. 1.

Other types of viscometers may be used, provided that the results are equivalent to those of the Ubbelohde viscometers specified above. However, in case of dispute, Ubbelohde viscometer shall be used.

A-3.10 Stainless Steel Sieve, with a nominal aperture size of 63 to 90 μm. or sintered glass filter funnel with 40 to 100 μm pore diameter.

A-3.11 Analytical Balance, accurate to 0·1 mg.

A-3.12 Stop-Watch, accurate to 0·1 s.

A-4 PROCEDURE

A-4.1 Cleaning the Viscometer

> **Warning** — Chromic acid solutions and other similar cleaning reagents are hazardous chemical. Avoid contact with the skin and inhalation of any vapours.

Clean the viscometers before use, after discordent readings and at intervals during regular use. To do this, let it stand for at least 12 h filled with a cleaning reagent, such as a chromic acid solution prepared by mixing 1 part by volume of concentrated sulphuric acid with 1 part of volume of a saturated solution of potassium dichromate. Remove the cleaning reagent, rinse the viscometer with distilled water followed by a suitable volatile solvent, such as freshly distilled acetone, and dry, for example, with a slow stream of filtered air.

After the determination, drain the viscometer, rinse with the solvent, then rinse with freshly distilled acetone or a similar volatile solvent, and dry as described.

If the next solution to be measured is of a polymer of the same type and of a similar viscosity, it is permissible to drain the viscometer, wash it with the solution to be measured, then fill it with the solution.

A-4.2 Selection of the Solvent

With poly (ethylene terephthalate), use phenol/1,2-dichlorobenzene as the solvent. With poly (butylene terephthalate) and copolymers of ethylene terephthalate and butylene terephthalate, use *m*-cresol as the solvent.

A-4.3 Determination of Inorganic Materials or Other Additives in the Sample

Contents of inorganic materials or other additives exceeding 0·5 percent (*m/m*) each shall be taken into account in the preparation of the test solution.

A-4.3.1 *Determination of the Content of Inorganic Materials*

If the sample contains inorganic materials, such as fillers or glass fibres, determine the content in an appropriate way. Report the procedure(s) applied.

A-4.3.2 *Determination of the Content of Other Additives*

If the sample contains other additives, such as polyalkenes or flame retardants, determine the content in an appropriate way. Report the procedure(s) applied.

A-4.4 Preparation of the Test Solution

Use one of the following procedures.

A-4.4.1 *Procedure A*

Weigh, to the nearest to 0·2 mg, a test portion, m_1, in grams, in the range:

$$\frac{0.5}{1-[(i+o)/100]} \pm 0.01$$

where

- i is the content of inorganic materials, expressed as a percentage by mass, in the sample, determined in accordance with **A-4.3.1**; and
- o is the content of other additives, expressed as a percentage by mass, in the sample, determined in accordance with **A-4.3.2**.

The corrections for i and o need only be applied if they exceed 0·5 percent (*m*/*m*) each.

Transfer the test portion to the volumetric flask, add 60 ml of solvent, stopper the flask and warm on the steam bath, with occasional stirring, until the polymer has disolved completely. On the steam bath, no degradation of polymer occurs. If, however, long waiting times are undesirable for other reasons, the dissolution, may be accelerated by applying continuous agitation, for example, by the magnetic stirrer.

Samples of highly crystalline poly (ethylene terephthalate), such as postcondensed moulding chips, which may have crystallinity around 65 percent will not dissolve on the steam bath, not even with continuous stirring. To dissolve such samples, the material shall be ground in the mill and dissolved at a temperature of 135°-140°C, some degradation of polymer occurs. Therefore, avoid heating times over 30 minutes.

After the dissolution, cool the flask and its contents to 25 ± 2° C, make up to 100 ml with solvent held at this temperature and mix well. If in dissolving a magnetic stirrer has been used, first remove it from the solution and rinse it with the make-up solvent, ensuring that all the washings enter the flask. The concentration 'C' of the polymer in the solution, expressed in g/ml, is computed as:

$$0{\cdot}01\left(1-\frac{i+o}{100}\right)m_1$$

A-4.4.2 *Procedure B*

Weigh, to the nearest 0·2 mg, a test portion, m_2, in the range 0·4 to 0·6 g. Transfer the test portion to the Erlenmayer flask. Add by burette, to the nearest 0·1 ml, a volume of solvent, in millilitres, equal to:

$$200\left(1-\frac{i+o}{100}\right)m_2$$

where i and o have the meaning as in **A-4.4.1**. The corrections for i and o need only be applied if they exceed 0·5 percent (*m*/*m*) each. The solvent added shall be at 25 ± 2°C.

Stopper the flask. Dissolve the polymer by heating the flask on the steam bath, or, if required, in the oil bath, following the procedures and observing the precautions mentioned in **A-4.4.1**.

After dissolution, cool the flask to room temperature.

A-4.5 Preparatory Operations

With phenol/1, 2-dichlorobenzene as the solvent, use a viscometer having capillary diameter of 0·73 mm ±2 %. With *m*-cresol, use a viscometer having capillary diameter of 1·03 mm ±2 % (*see* Fig. 1).

Filter the test solution through the screen to sintered glass filter into tube *L* of the viscometer (*see* Fig. 1). The volume should be such that, after draining, the level lies below the filling marks. Carry out this filling operation away from the thermostatic bath to prevent contamination of the bath in case of accidental spills.

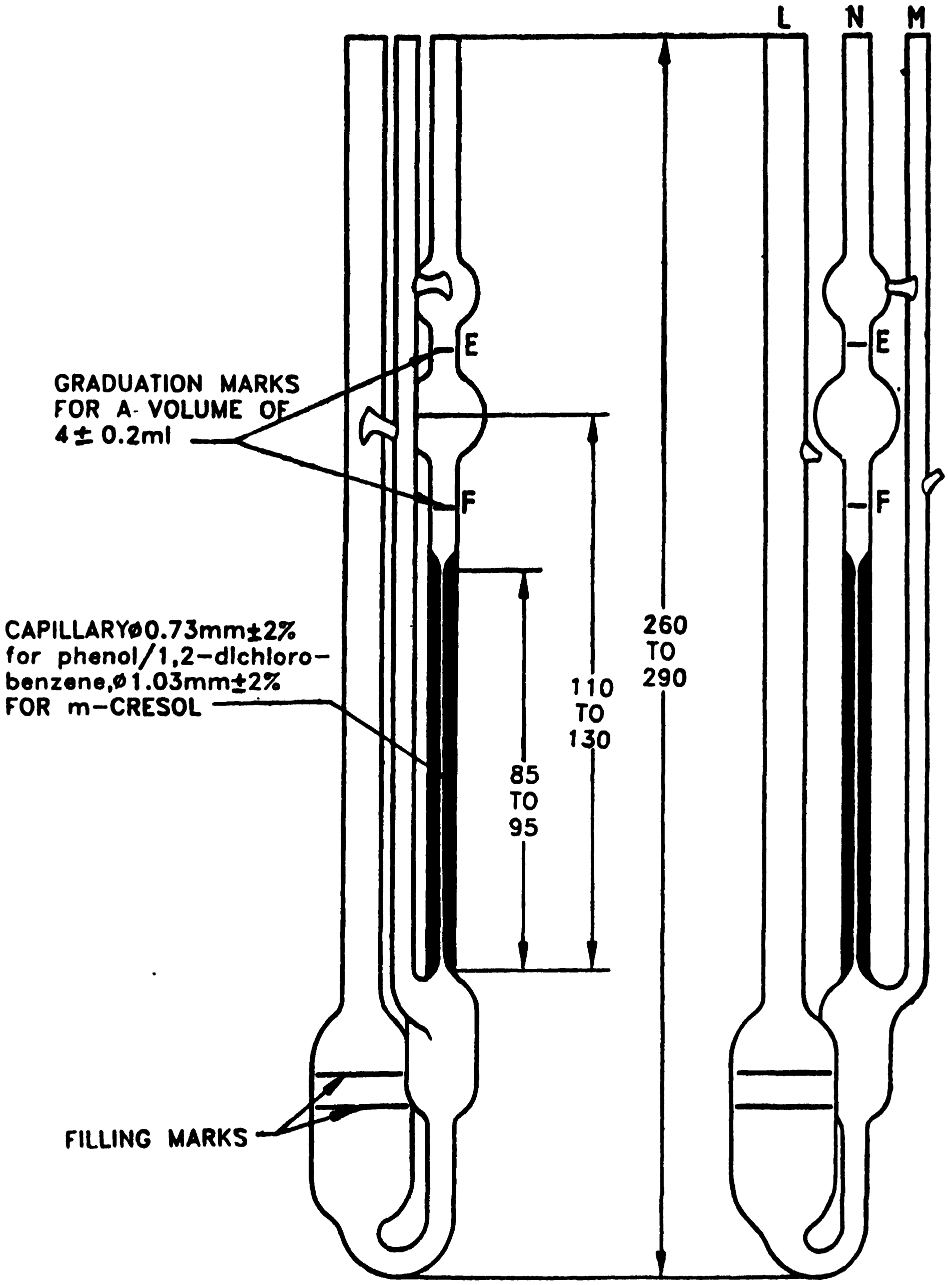
Mount the viscometer in the thermostatic bath, ensuring that tube *N* is vertical and that the upper graduation mark, *E*, is at least 30 mm below the surface of the bath liquid. Allow at least 15 minutes for the charged viscometer to attain the temperature of the bath (25 ± 0·05° C).

A-4.6 Measurement of Flow Times

Close tube *M* and apply suction to tube *N* or pressure to tube *L* until the liquid reaches a level about 5 mm above mark *E*. Hold the liquid at this level by closing tube *N*. Open tube *M* so that the liquid drains away from the lower end of the capillary tube.

When the liquid is clear of the capillary end and of the lower end of the tube *M*, open tube *N*. Measure the flow time to an accuracy of 0·2 s as the time taken for the lowest point of the meniscus to pass from the top edge of mark *E*, to the top edge of Mark *F*. For polymer solution containing small amounts of divided pigments, for example, carbon black, it may be necessary to view the top of the meniscus.

NOTE — The opening and closing of the tubes may conveniently be carried out by means of taps or clips on clean flexible tubing attached to the end of the tubes.



All dimensions in millimetres.

FIG. 1 UBBELOHDE VISCOMETER

Repeat the measurement of the flow times immediately, without emptying and recharging until two successive flow times agree within 0·25 percent. Take the mean of these two flow times as the flow time of the test solution.

Determine the mean flow time of the solvent in the same viscometer and in the same manner as that of the test solution. It suffices to measure this time once for each series of determinations. However, measure the time at least once each day the solvent is used. If two successive determinations of the mean flow time differ by more than 0·4 s, clean the viscometer.

A-5 EXPRESSION OF RESULTS

Calculate the Relative Viscosity (RV) according to the formula:

$$RV = \frac{t_1}{t_0}$$

where

t_1 = flow time, in seconds, of the test solution; and

t_0 = flow time, in seconds of the solvent.

With each polymer sample, carry out at least two determinations of *RV*, each time using a fresh solution, until two successive values differ by not more than 3 percent. Report the mean of these two values rounded off to the nearest unit, as the relative viscosity of the sample.

ANNEX B

[*Clause* 5.4.3, and *Table* 1, *Item* (x)]

DETERMINATION OF MOISTURE CONTENT

B-1 GENERAL

The method for determination of the water content of polyethylene terephthalate and polybutylene terephthalate granules is applicable for the determination of water content in the range 0·002 to 0·05 percent (*m*/*m*).

Water content is of importance in connection with the processing of the material, during which it should be below a few hundredths of a percent to prevent degradation.

The method is not applicable to polyethylene terephthalate and polybutylene terephthalate samples containing volatile compounds, other than water, in amounts contributing significantly to the vapour pressure at room temperature. The error introduced by small amounts of acetaldehyde usually present in dried polyethylene terephthalate granules is considered to be acceptable. Checks for the presence of larger amounts of volatile compounds shall be carried out periodically, for example, by gas chromatography. Such checks are particularly required for new types or grades of material.

B-2 OUTLINE OF THE METHOD

A test portion is heated to 200 ± 5°C in a closed space under a high vacuum, thus ensuring complete evaporation of the water. The resulting pressure increase, to which the water content is proportional, is measured. The water content is calculated by reference to a calibration curve prepared using a hydrate with a known water content, such as sodium molybdate, dihydrate, which loses its water under the conditions of the test.

B-3 REAGENT

Sodium Molybate, Dihydrate ($Na_2M_oO_4 2H_2O$), of recognized analytical quality.

NOTE — Other hydrates which lose their water of crystallization under the conditions of test, such as barium chloride dihydrate ($BaCl_2 2H_2O$), may also be used.

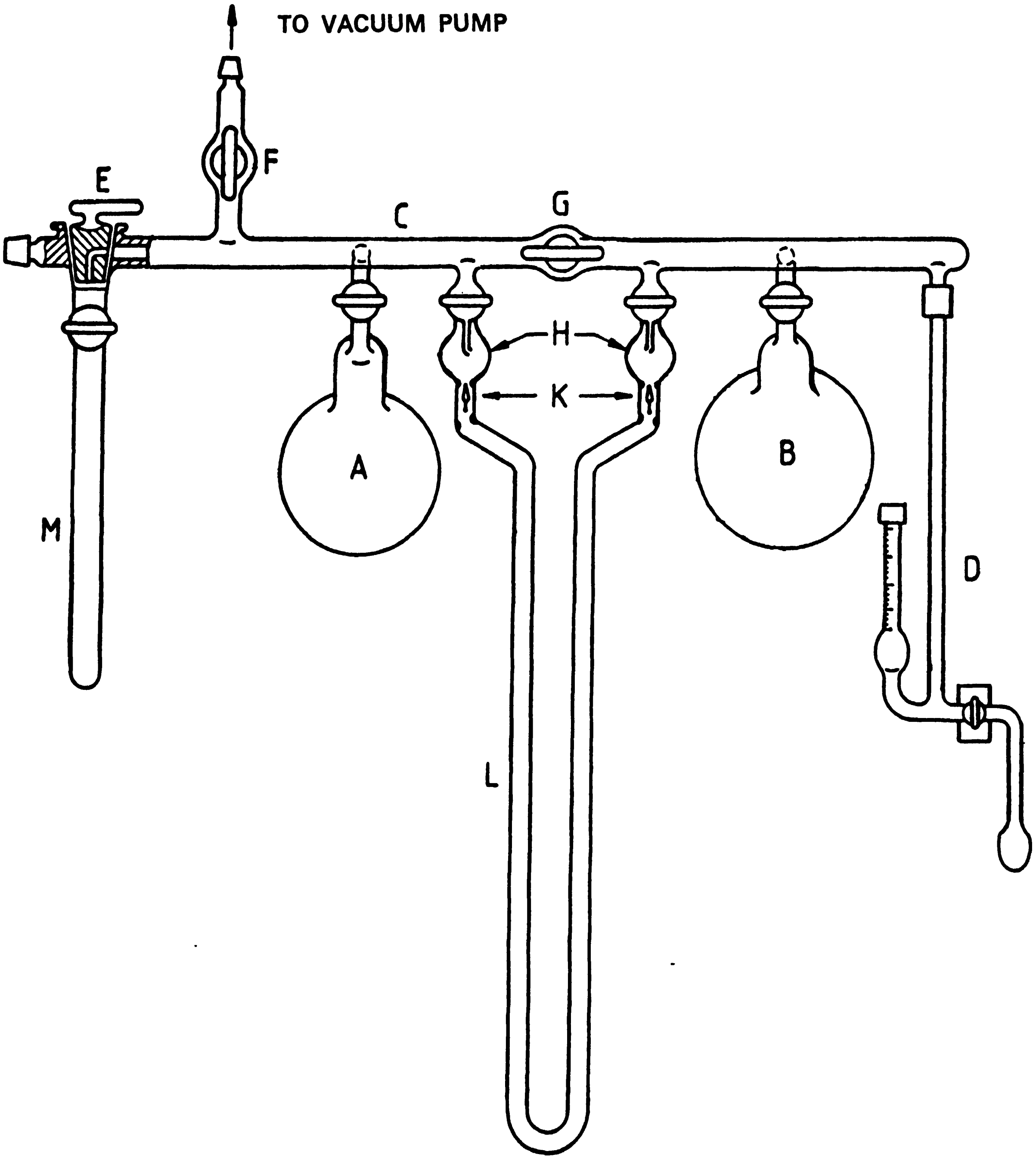
B-4 APPARATUS

Ordinary laboratory apparatus.

B-4.1 Measuring Apparatus

The use of a measuring apparatus as described below is recommended. The equipment is shown diagrammatically in the Fig. 2. It is an all-glass system with vacuum-tight connections, preferably in the form of spherical joints.

Bulbs (*A*) and (*B*) have volumes of 0·5±0·05 litre and at least 1 litre, respectively.



Key

A	Bulb, 0·5 ± 0·05 litre	*H*	Splash heads
B	Bulb, > 1 litre	*K*	Check valves
C	Connecting tube	*L*	Oil manometer
D	High-vacuum gauge	*M*	Sample tube
E, F, G	Stopcocks		

FIG. 2 APPARATUS FOR DETERMINATION OF WATER CONTENT

The bulbs are connected to a tube (C), which is connected at one end to a high-vacuum gauge (*D*), and at the other end to a sample tube-adapter fitted with a stopcock (*E*). The tube (*C*) carries a connection to a vacuum pump fitted with a stopcock (*F*) and is fitted with a stopcock (*G*) to separate the bulbs. On both sides of the stopcock (*G*) the tube is connected via splash heads (*H*) and check valves (*K*) to a U-tube oil manometer (*L*), the legs of which have a length of at least 350 mm. The sample tube (*M*) shall be made of heat resistant glass. The sample tubes in a set shall not differ in volume by more than 5 ml.

NOTES

1 The use of an apparatus of a different design is allowed, provided that the repeated requirements in B-6.2.2 can be met.

2 Silicone oil is suitable for filling the manometer.

B-4.2 Heating Device

An electric oven or any other suitable device may be used to heat the sample tube to 200 ± 5°C. The arrangement of the equipment shall preferably be such as to allow easy installation and removal of the heating device.

B-5 SAMPLING

Quickly fill a dry container with a representative sample of the test material and immediately close it to minimize moisture uptake from the atmosphere.

NOTE — It is desirable to pre-dry the container in an oven and to cool it above a suitable water absorbent, for example, blue silica gel.

B-6 PROCEDURE

B-6.1 Leakage Check

Check the apparatus for leakage as follows.

B-6.1.1 Fix a dry, empty sample tube, which does not need to be heated during the check, to the apparatus. Turn stopcock (*E*) to connect the sample tube to tube (*C*) and turn stopcock (*G*) to connect bulbs (*A*) and (*B*).

B-6.1.2 Evacuate the system to a pressure of less than 100 Pa (100 Pa = 1 mbar = 0·7 mm Hg) and close stopcocks (*F*) and (*G*).

B-6.1.3 After 1 hour, check that the pressure is still less than 100 Pa and that the pressure difference indicated by the manometer is less than 2 mm of oil. If these requirements are not met, check for leaks and repeat the test.

B-6.1.4 Carry out checks as frequently as necessary to ensure air-tightness during the determinations.

NOTE — When the oil in the manometer is replaced evacuation of the apparatus for a few hours may be required for de-aeration of the new oil.

B-6.2 Determination

B-6.2.1 Quickly pour a volume of the test sample corresponding to a mass of 12 to 18 g into a dry sample tube and fix the tube to the apparatus. (*see* Note 1.)

Turn stopcock (*E*) to connect the sample tube to tube (*C*) and turn stopcocks (*G*) to connect bulbs (*A*) and (*B*).

Evacuate the system to a pressure of less than 100 Pa and close stopcocks (*F*) and (*G*) (*see* Note 2).

Position the heating device, previously heated to 200 ± 5°C, around the sample tube and heat the tube at this temperature for 50 minutes or until the pressure difference indicated by the oil manometer remains constant up to 1 mm for 5 minutes (*see* Note 3).

After 50 minutes, or when the pressure difference remains constant, read the pressure difference to the nearest mm.

Discontinue the heating of the sample tube, open stopcock (*G*) and break the vacuum in the sample tube by turning stopcock (*E*).

Allow the sample tube to cool and weigh its contents to the nearest 0·01 g.

NOTES

1 Initially, the test portion is measured by its volume to minimize moisture uptake from the atmosphere. The weighing is carried out after heating.

2 When testing powder materials, carry out the evacuation slowly. It is recommended that the test portion in the tube be covered by a thin layer of glass wool. Pre-dry the glass wool in an oven and cool and store it above a suitable water absorbent such as a blue silica gel.

3 For the unknown samples, high moisture contents may not be excluded. Therefore, constantly observe the manometer for the first 10 minutes of the test and, if the pressure becomes too high, open stopcock (*G*) and repeat the test with a smaller test portion.

B-6.2.2 Carry out two determinations on each sample. If the results differ by more than 0·005 percent (*m*/*m*), check for leaks (*see* **B-6.1**) and carry out two further determinations.

B-6.3 Calibration

B-6.3.1 Weigh at least five test portions of sodium molybdate dihydrate, about 30 to 40 mg each, and place them in clean, dry sample tubes.

Carry out the procedure specified in **B-6.2.1** with each portion of sodium molybdate dihydrate. The length of the heating period may be reduced from 50 to 15 minutes.

B-6.3.2 Calculate the calibration factor f, corresponding to the mass of water, in grams required to produce a pressure difference of 1 mm of oil, using the equation:

$$f = \frac{m \times w}{\Delta p}$$

where

m = mass in g, of the test portion of sodium molybdate dihydrate;

w = water content, in g per gram, of the sodium molybdate dihydrate;

Δp = pressure difference, in mm of oil, indicated by the manometer.

If a hydrate other than sodium molybdate dihydrate is used for the calibration, adapt the mass of the test portions and the value of w correspondingly.

Calculate the factor f as the average of the values obtained with the different test portions. In this calculation, disregard results that differ by more than 5 percent from the average of the other results.

NOTES

1 When a new batch of sodium molybdate dihydrate is used, check the water content by weighing, drying for 1 h at 200°C and reweighing.

2 Do not use water, as such, for the calibration since the amounts required would be too small for weighing with sufficient accuracy.

B-7 EXPRESSION OF RESULTS

The water content, expressed as a percentage by mass is given by the formula:

$$\text{Water content, percent by mass} = \frac{f \Delta p}{m} \times 100$$

where

f = calibration factor, determined as described in **B-6.3**;

Δp = pressure difference, in mm of oil indicated by the manometer;

m = mass, in g, of the test portion of poly (alkylene terephthalate).

B-8 TEST REPORT

The test report shall include the following particulars:

a) Reference to this Indian Standard;

b) Completete identification of the test sample; and

c) The individual test results and the average value of the two determinations to the nearest 0·001 percent (*m/m*).

ANNEX C

(*Clause* 5.6.1)

DETERMINATION OF THE BURNING BEHAVIOUR OF VERTICAL PLASTICS SPECIMENS IN CONTACT WITH A SMALL FLAME IGNITION SOURCE

C-1 GENERAL

C-1.1 This is a small-scale laboratory screening procedure for comparing the relative burning behaviour of vertically oriented plastic specimens exposed to a low energy level ignition.

C-1.2 This method of test determines the afterflame/afterglow times and damaged length of specimens. It is applicable to solid and cellular materials having an apparent density of not less than 250 kg/m^3. This method is not applicable for materials that spring away from the applied flame without igniting.

C-1.3 The classification system described is intended for quality assurance and the preselection of component materials for products. This system is not intended to assess the fire behaviour of building materials, furnishings, complete items of equipment or finished parts.

C-2 OUTLINE OF THE METHOD

A test specimen bar is supported vertically by one end and the free end is exposed to a specified gas flame. The burning behaviour of the bar is assessed by measuring the afterflame/afterglow times.

C-3 SIGNIFICANCE OF TEST

C-3.1 Test made on a material under the conditions specified may be of considerable value in comparing the relative burning behaviour of different materials, in controlling manufacturing processes or in assessing any change in burning characteristics prior to, or during, use. The results obtained from this method are dependent upon the shape, orientation and environment surrounding the specimen and the conditions of ignition. Correlation with performance under actual service conditions is not implied.

C-3.2 Results obtained in accordance with this method shall not be used to describe or appraise the fire hazard presented by a particular material or shape under actual fire conditions, unless used as one element of a fire risk assessment that takes into account all of the factors that are pertinent to the assessment of the fire hazard in a particular end use for the material. Assessment for fire hazard requires consideration of such factors as fuel contribution, intensity of burning (rate of heat release) products of combustion and environmental factors such as the intensity of source, orientation of exposed material and ventilation conditions.

C-3.3 Burning behaviour as measured by this test method, is affected by such factors as density, any anisotropy of the material and the thickness of the specimen.

C-3.4 Certain materials may shrink from the applied flame without igniting. In this event test results are not valid and additional test specimens are required to obtain ten valid tests. If the test specimens continue to shrink from the applied flame without igniting, these materials are not suitable for evaluation by this method of test.

C-3.5 The burning behaviour of some plastic materials may change with time. It is accordingly advisable to make tests before and after ageing by an appropriate procedure. The preferred ageing conditions shall be 7 days at 70°C. However, other ageing times and temperatures may be used by agreement between the interested parties and shall be noted in the test report.

C-4 APPARATUS

C-4.1 Laboratory Fume Hood (Cupboard), having an inside volume of at least 0·5 m^3, shall be used when testing the specimens. The chamber shall permit observation and shall be draught-free while permitting normal thermal circulation of air past the specimen during burning. For safety and convenience, it is desirable that this enclosure (which may be completely closed) be fitted with an evacuation device, such as an exhaust fan, to remove products of combustion which may be toxic. However, it is important to note that the device shall be turned off during the actual test and started again immediately after the test to remove the products of combustion.

NOTE — The amount of oxygen available to support combustion is naturally important for the conduct of these flame tests. For tests conducted by this method when burning times are protracted, chamber sizes less than 1 m^3 may not provide accurate results.

C-4.2 Laboratory Burner, A bunsen burner having a tube length of 80 to 100 mm and an inside diameter of $9{\cdot}4\,^{+1{\cdot}6}_{-0{\cdot}0}$ mm. The tube shall not be equipped with an end-attachment such as a stabilizer.

C-4.3 Ring Stand, With Clamps or the Equivalent, adjustable for positioning of the specimen.

C-4.4 Timing Device, accurate to 1 second.

C-4.5 Measuring Scale, graduated in mm.

C-4.6 Supply of Technical Grade Methane Gas, with regulator and meter for uniform gas flow. Other gas mixtures having a heat content of approximately 37 MJ/m^3, have been found to provide similar results.

C-4.7 Desiccator, containing anhydrous calcium chloride or other drying agent.

C-4.8 Conditioning Room or Chamber, capable of being maintained at 27 ± 2°C and a relative humidity of 65 ± 5 percent.

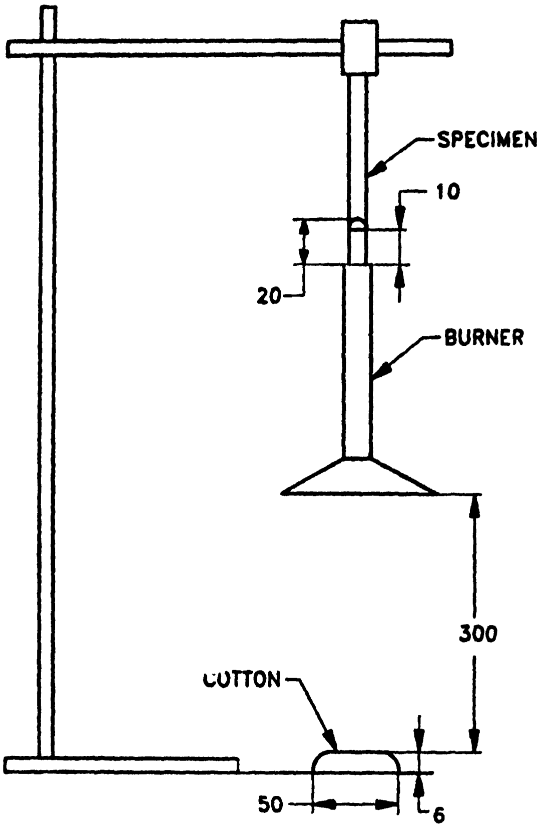
C-4.9 Complementary Apparatus (*see* Fig. 3).

C-4.10 Dry Absorbent Surgical Cotton

C-4.11 Full-Draught Air-Circulating Oven, minimum of 25 air changes/hour, capable of being maintained at 70 ± 1°C or other agreed temperature.

C-5 SPECIMENS

C-5.1 All specimens shall be cut from a representative sample of the material (sheets or from



All dimensions in millimetres.

FIG. 3 VERTICAL BURNING-BEHAVIOUR TEST APPARATUS

end-products), or shall be cast or injection, compression or transfer moulded to the necessary form. After any cutting operation, care shall be taken to remove all dust and any particles from the surface; cut edges shall have a smooth finish.

C-5.2 Standard bar specimens shall be 125 ± 5 mm long, 13·0 ± 0·3 wide and 3·0 ± 0·2 mm thick. Other thicknesses may be used by agreement between the interested parties and, if so shall be noted in the test report.

NOTE — Tests made on specimens of different thicknesses or density are not comparable and tests made in different directions of anisotropy may also not be comparable.

C-5.3 A minimum of 26 bar specimens shall be prepared. It is advisable to prepare additional specimens in the event that the situation described in **C-3.4** is encountered.

C-6 CONDITIONING

C-6.1 Unless otherwise required by the material specification, the following shall apply.

C-6.1.1 Two sets of 5 bar specimens shall be preconditioned for at least 48 h at 27 ± 2°C and 65 ± 5 percent relative humidity.

C-6.1.2 Two sets of 5 bar specimens shall be preconditioned for 168 h at 70 ± 1°C and then cooled in a desiccator (**C-4.7**) for at least 4 h at ambient temperature.

C-6.1.3 All specimens shall be tested in a standard laboratory atmosphere of 27 ± 2°C and 65 ± 5 percent relative humidity.

C-7 PROCEDURE

C-7.1 Clamp the specimen from the upper 6 mm of its length with the longitudinal axis vertical so that the lower end of the specimen is 300 mm above a horizontal layer of dry absorbent surgical cotton (50 × 50 mm) thinned to a maximum uncompressed thickness of 6 mm (*see* Fig. 3).

C-7.2 Adjust the burner to produce a blue flame 20 ± 1 mm high. The flame shall be obtained by adjusting the supply and air ports of the burner until an approximate 20 mm yellow-tipped blue flame is produced. Increase the air supply until the yellow tip disappears. Measure the height of the flame again and adjust it if necessary.

C-7.3 Place the flame of the burner centrally under the specimen, so that the top of the burner is 10 mm below the lower end of the specimen, and allow it to remain there for 10 seconds. Withdraw the burner to a distance at least 150 mm away and simultaneously start the timing device. Note the afterflame time t_1, in seconds. If the specimen drips molton or flaming material during flame application, the burner may be tilted to an angle of 45° to avoid material dripping into the tube of the burner. However, the 10 mm distance shall be maintained between the major portion of the specimen and the tilted burner.

C-7.4 When afterflaming of the specimen ceases, immediately place the flame of burner again under the specimen. After 10 seconds turn off the burner and note the afterflame t_2 and afterglow t_3 times of the specimen.

C-7.5 The test procedure shall be conducted on at least five specimens.

C-8 EXPRESSION OF RESULTS

C-8.1 Calculate the total afterflame time t_{fi} in seconds, for an individual specimen, using the formula:

$$t_{fi} = t_1 + t_2$$

where

t_1 = first afterflame time, in seconds;

t_2 = second afterflame time, in seconds; and

i = specimen number.

C-8.2 For each set of five specimens from a given preconditioning treatment, calculate the total set afterflame time t_{fs}, in seconds, using the formula:

$$\sum_{i=1}^{i=5} t_{fi}$$

where i and t_{fi} are as defined in **C-8.1**.

C-8.3 Calculate the combustion time t_{ci} in seconds, for an individual specimen, using the formula:

$$t_{ci} = t_2 + t_3$$

where

i and t_2 are as defined in **C-8.1**; and

t_3 = afterglow time, in seconds.

C-9 CATEGORIES OF BURNING BEHAVIOUR

The behaviour of the specimens shall be classified in one of the categories given in Table 2 (FV = Flaming Vertical specimen) determined by selecting the appropriate column, using test results to answer the conditional questions posed.

C-10 TEST REPORT

The test report shall include the following particulars:

a) Complete identification of the product tested, including the manufacturer's name, number or code;

b) The thickness to the nearest mm, of the test specimen;

c) The direction of any anisotropy relative to the test specimen dimensions;

d) Conditioning treatment;

e) Any prior treatment before testing, other than cutting, trimming and conditioning;

f) Classification according to the category code designation specified in C-9.

Table 2 Categories of Burning Behaviour

(*Clause* C-9)

Sl No.	Conditions	Category (*see* Note 1)			
(1)	(2)	(3)	(4)	(5)	(6)
i) If	: Any individual specimen total afterflame t_{fi}	$\leqslant$10 s	$\leqslant$30 s	$\leqslant$30 s	>30 s
ii) And	: Total set afterflame time t_{fs}	$\leqslant$50 s	$\leqslant$250 s	$\leqslant$250 s	>250 s
iii) And	: Any individual specimen combustion time after the second flame application t_{ci}	$\leqslant$30 s	$\leqslant$60 s	$\leqslant$60 s	>60 s
iv) And	: Afterflame or afterglowing up to the specimen holding clamp	No	No	No	Yes
v) And	: Cotton indicator ignited by flaming particles or drops	No	No	Yes	Yes or no
vi) Then	: The category is	FV - 0	FV - 1	FV - 2	(*see* Note 2)

NOTES

1 If only one specimen from a set of five specimens for a given preconditioning treatment does not comply with the requirements for a category, another set of five specimens subjected to the same preconditioning shall be tested. All specimens from the second set shall comply with the appropriate requirements for the category.

2 The material may not be categorized by this method.

ANNEX D

(*Clause* 7)

SAMPLING OF POLYALKYLENE TEREPHTHALATES (PET & PBT)

D-1 GENERAL

D-1.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

D-1.2 Samples shall not be taken in an exposed place.

D-1.3 The sampling instrument wherever applicable, shall be made of stainless steel or any other suitable material on which the material shall have no action. The instrument shall be clean and dry.

D-1.4 Precautions shall be taken to protect the samples, and material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

D-1.5 The sample shall be placed in a suitable, clean, dry, and airtight metal containers on which the material has no action. The sample containers shall be of such a size that they are almost completely filled by the sample.

D-1.6 Each sample container shall be sealed airtight with a stopper after filling and marked with full details of sampling, such as type of sample, date of sampling, and the month and year of manufacture of material.

D-1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

D-2 SCALE OF SAMPLING

D-2.1 Lot

In a single consignment all the packages of the same class, same type, same form and belonging to the same batch of manufacture shall be grouped together to constitute a lot. If a consignment is known to consist of packages belonging to different batches of manufacture or different forms, the packages belonging to the same batch of manufacture and same form shall be grouped together and each such group shall constitute a lot.

D-2.2 For acertainining the conformity of the material to the requirements of this specification, samples shall be tested from each lot separately. The number of packages to be sampled shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 3.

Table 3 Scale of Sampling

Number of Packages in the Lot	Sample Size
(1)	(2)
Up to 50	3
51 to 150	4
151 to 300	5
301 to 500	7
501 and above	10

D-2.2.1 These packages shall be selected at random from the lot and in order to ensure the randomness of selection, procedure given in IS 4905 : 1968 may be followed.

D-3 PREPARATION OF TEST SAMPLES

From each of the packages of material selected, small portions of material shall be drawn with the help of suitable sampling instrument. The total quantity of material collected from each package shall be sufficient to test all the requirements given in 5 of the standard.

D-4 NUMBER OF TESTS

Tests for determining all the requirements given in 5 shall be carried out on the individual test samples.

D-5 CRITERIA FOR CONFORMITY

The lot shall be declared as conforming to the requirements of this sepcification if all the test results on individual samples meet the relevant specification requirements.

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Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 84 99, 37 85 61, 37 86 26, 37 86 62
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